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QUALITY ASSURANCE SAMPLING PLAN
FOR THE COLLECTION OF AIR AND SOIL SAMPLES
FROM RESIDENTIAL AREAS NEAR THE CERTAIN-TEED/MALINE CREEK SITE
FOR ASBESTOS ANALYSIS

(BELLEFONTAINE NEIGHBORS, ST. LOUIS COUNTY, MISSOURI)

Site:	Maline Creek
ID #	MoD980631162
Break:	2.3
Other:	EFE
	10-94

U.S. EPA REGION VII
EMERGENCY PLANNING AND RESPONSE BRANCH

and

ECOLOGY AND ENVIRONMENT, INC.
TECHNICAL ASSISTANCE TEAM

October, 1994

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APPROVED:

[Signature]
On-Scene Coordinator

12/22/94
Date

[Signature]
Peer Reviewer

12/22/94
Date

Chief, Emergency Planning & Response Branch

Date

Regional Quality Assurance Officer

Date

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I. INTRODUCTION

A. Site Location

The Certain-Teed/Maline Creek site is located at 600 St. Cyr Road in Bellefontaine Neighbors, Missouri. Bellefontaine Neighbors is a suburban city in north St. Louis County. The geographic coordinates of the site are 38°44'03" N latitude and 90°13'12" W longitude. A site location map is included as attachment A to this plan.

B. Site Description

The site consists of the former Certain-Teed Transite Pipe Plant property at 600 St. Cyr Road and the former GAF Transite Plant property at 9215 Riverview Boulevard, adjacent to the former Certain-Teed property to the south-southeast. Both Certain-Teed and GAF manufactured asbestos containing transite pipe and sheeting and used the field between the two facilities as an open dump for scrap materials (Attachment B). The area around the properties is industrial/residential and is in the city limits of Bellefontaine Neighbors and Riverview, Missouri. Maline Creek flows south-southeast along the southern boundary and eventually empties into the Mississippi River approximately three-fourths of a mile from site. The area directly south-southeast of Maline Creek along the site is a residential subdivision and there is a nursing home 350 feet northwest of the site.

C. Site History

Certain-Teed Corporation manufactured asbestos-cement pipe at this site from the mid 1920s until May 11, 1979, when manufacturing operations ceased. The neighboring GAF Transite Plant also ceased operation sometime in 1979. Up until that time, both facilities reportedly used the land between their plants as an open dump for scrap asbestos and settled solids from process wastewater. In February 1979, both companies hired the same consulting engineering firm, Reitz & Jens, to begin preparing closure plans for Certain-Teed and GAF to minimize the potential for adverse environmental impact and to comply with Missouri Solid Waste Management Law. Subsequent plans approved by the Missouri Department of Natural Resources (MDNR) included reworking the material to an acceptable slope, applying an earthen cover of at least 12 inches, seeding the site to establish vegetative growth, and constructing a rock

covering on the creek slope to prevent erosion.

A site inspection conducted by MDNR on May 13, 1980, confirmed that the site was in basic conformance with the approved closure plans, however it was noted that broken pieces of asbestos-containing pipe were scattered along the undisturbed creek bank upstream of the rip-rap work area and south of the former Certain-Teed facility. This condition was not determined to pose a significant threat due to the wooded nature of the creek bank at that time. The Certain-Teed Corporation sold the property to the current owner, P.G. Investments, in September 1981. P.G. Investments, owned and operated by Phillip and Gerald Kootman, subsequently opened Branch Metal Processing Company at the site. In January 1982, pipe material became visible along the creek bank after the Metropolitan St. Louis Sewer District (MSD) conducted tree and brush removal along the creek to facilitate future creek channelization efforts. This left the material subject to sloughing and weathering with stream flow fluctuations. MDNR recommended at that time that any removal and stabilization efforts be coordinated with MSD.

In May 1982, MSD proposed a cleanup of the creek bank. MDNR approved the plan with the condition that the waste be disposed at an approved sanitary landfill. The cleanup began in August 1982, with several loads of scrap asbestos containing material hauled to West Lake Sanitary Landfill in Bridgeton, Missouri. According to MDNR reports, when these efforts ceased there was still approximately 1000 square feet of scrap asbestos pipe visible along the upper portion of the creek bank.

The EPA Environmental Monitoring and Compliance Branch (EMCM) conducted inspections of the former Certain-Teed and GAF facilities in May and June of 1988 respectively. Exposed transite pipe and board was observed along the creek bank and on the surface near the covered waste piles at both facilities and transite pipe was observed in the creek bed along the Certain-Teed property. Samples of the exposed materials collected during these inspections indicated the materials contained up to 25% chrysotile and 15% crocidolite asbestos. Followup site assessment activity was conducted at the site in March and September 1992, by the Ecology & Environment, (E & E) Inc., Technical Assistance Team (TAT) following a congressional inquiry to EPA initiated by a citizen complaint. Further sampling was conducted and photographic and video documentation of the site was produced. Sample results from this effort indicated exposed insulation, transite pipe, and sheeting materials containing up to 85% chrysotile and 15 % crocidolite asbestos. The exposed materials appeared to be weathering and becoming more friable and scrap materials were observed accumulating in the creek bed as the pieces were dislodged from the creek bank through erosional processes.

During the flood event in July and August of 1993, swelling of the Mississippi River caused a back up of Maline Creek to such an extent that flooding of the common area along the south bank of the

creek adjacent to the subdivision south of site occurred. In addition approximately 70 homes were flooded to varying degrees during the peak crest period. The peak crest on the Mississippi River in St. Louis occurred on August 1, 1993, with a crest stage of 49.6 feet. Approximately 20 of the affected homes are scheduled for buyout by the Federal Emergency Management Agency (FEMA). More homes were eligible for buyout however the residents refused. This flood event potentially transported asbestos fibers from the site, increasing the potential for asbestos contamination in the affected areas above and beyond that which may have been present prior to the flood.

II. OBJECTIVES

A. Objectives of Sampling Effort

The primary objective of this proposed sampling effort is to provide a rapid assessment of the potential threat from exposure to asbestos fibers to residents living in the subdivision near the site. The target population in this assessment is the residences near the area affected by the flooding in 1993, however the sampling will be conducted with the assumption that contamination by asbestos fibers at these residences may have been occurring in this area due to entrainment from the site for many years prior to the flood event.

B. Scope of Work

To achieve the aforementioned objectives for this sampling effort a network of personal sampling pumps will be set up to collect air samples for asbestos analysis near the residences affected by flooding in August, 1993. Soil samples will also be collected for asbestos analysis from selected locations. In order to determine what effect the flood may have had in spreading asbestos contamination off site several remote and background sample locations will be selected away from the area affected by the flooding. The air samples will be collected with personal sampling pumps in accordance with the National Institute for Occupational Safety and Health (NIOSH) method 7402 and with the assistance of an Asbestos Hazard Emergency Response Act (AHERA) certified air monitoring technician.

C. Data Quality Objectives

The data quality objective for this sampling effort is to provide data to give a rapid assessment of the potential threat from exposure to asbestos fibers to residents living near the Certain-Teed/Maline Creek site. Definitive identification and quantitation of the asbestos fibers in all samples will meet quality assurance level two (QA2) objectives and provide a health

and safety assessment for the site.

III. PROPOSED FIELD ACTIVITIES

A. Sampling Rationale/Methods

As mentioned previously, the air samples will be collected according to NIOSH method 7402 and in accordance with 29 CFR 1910.1001. An AHERA certified air monitoring technician will be subcontracted to assist with the air sampling network design and sample collection. The network will be set up in the flood zone and at remote and background locations upwind and away from the site. As specified in the method, personal sampling pumps will be set up to run for eight hours at a rate of two liters per minute giving an approximate total sample volume of 960 liters. Each pump will be calibrated before and after use with a representative filter cassette installed. The collection medium or filter cassette will be the prescribed 25-mm diameter cassette with an open-faced 50-mm electrically conductive extension cowl and mixed cellulose ester filter membrane. The filter cassettes will be set at one meter above the ground during sample collection. A total of 12 air samples, including two blank samples as specified in 29 CFR 1910.1001 and one collocated sample, will be submitted to the contracted laboratory for transmission electron microscopy (TEM) asbestos analysis following NIOSH method 7402.

Surface soil samples will be collected from all air sampling locations and from selected other locations in the flood zone, remote, and background locations following E & E Standard Operating Procedures (SOP) for Soil Sampling Geotech 5.17, January 1990. The soil samples collected in association with the air sample locations will be composite samples consisting of four aliquots collected approximately 10 feet north, south, east, and west of the pump location. The remaining soil samples will be grab samples collected from selected locations. The samples will be packaged in 8-ounce glass jars. A maximum of 30 soil samples will be submitted to the contracted laboratory for TEM asbestos analysis.

A field logbook will be kept documenting all activity during this sampling. Sample documentation and management in the field will be conducted according to the following standard operating procedures:

- 2130.2A Field Chain of Custody for Environmental Samples
- 2130.3A Identification, Documentation and Tracking of Samples

B. Sampling Equipment

- 10 Gilian personal sampling pumps with flexible connecting tubing
- 1 Gilibrator primary standard airflow calibrator
- 12 25-mm x 50-mm electrically conductive filter cassettes

with mixed cellulose ester filter membranes

- compass
- survey flags
- 100' and 300' tapes
- stainless steel sampling spoons
- aluminum pie pans
- powder free vinyl surgical gloves
- 8-ounce glass sample jars
- poultry bags
- 38" x 60" poly bags
- trash bags
- paper towels
- tap water
- field sheets, sample tags, custody seals, chain-of-custody forms
- duct tape, strapping tape, clear tape
- static free packing material
- cooler for sample storage and shipment
- Level C personal protective equipment (PPE)
- 35-mm camera and film
- logbook

C. Decontamination Procedures

Dry decontamination and disposal of all PPE and expendable sampling equipment is suggested. PPE will be rendered useless and bagged for disposal. Equipment and instrumentation will be wiped down with moist towelettes and wiped dry.

IV. LOGISTICS

A. Personnel Requirements/Protective Equipment

Two TAT personnel will be required to conduct the soil sampling and to assist the subcontracted AHERA certified air monitoring technician with the air sampling. The EPA On-scene Coordinator (OSC) for this project is Don Hamera. All sampling will be conducted in level C PPE with an air-purifying respirator and hooded tyvek coveralls. Latex boot covers and inner gloves will be utilized and sealed with duct tape to the tyvek coveralls. A new pair of powder free vinyl surgical gloves will be donned for each sample collected to minimize the potential for cross contamination of samples.

B. Schedule

A meeting between EPA, MDNR, and local personnel regarding the site and access has been scheduled for Thursday October 27, 1994. The sampling effort has been tentatively scheduled to follow this meeting on Tuesday November 1 and Wednesday November 2, 1994. The

sampling will be postponed if it is raining or conditions are wet.

C. Access

Permission to access the former Certain-Teed property will be obtained through EPA at the meeting on October 27, 1994. Permission to access the residences and remote locations for air and soil sampling will be acquired at this same time by the OSC.

D. Media/Public Inquiries

All inquiries concerning the site and the sampling effort will be referred to the OSC or the EPA Region VII Office of Public Affairs for response.

V. ANALYTICAL METHODS

A. Analytical Procedures

Both the air and soil samples will be analyzed by transmission electron microscopy (TEM) at a National Institute for Standards and Technology (NIST) accredited laboratory. As mentioned previously, the air samples will be collected and analyzed according to NIOSH method 7402 and in accordance with 29 CFR 1910.1001 guidelines. The data will be reported in fibers per cubic centimeter of air (f/cc). Soil samples will be analyzed by the TEM modified Chatfield method with the data reported in percentage by fiber species. Holding time on the samples is indefinite.

B. Method Detection Limits

The estimated method detection limit for TEM analysis for asbestos in air is .005 fibers per cubic centimeter. A structure must be longer than five microns and have at least a 3 to 1 length to diameter ratio to be counted as a fiber. An action level of 0.1 fiber per cubic centimeter as an 8-hour time weighted average (TWA) has been established as the concentration above which employers must initiate compliance activities. The Occupational Safety and Health Administration (OSHA) permissible exposure limit (PEL) for asbestos is an 8-hour TWA of 0.2 fiber per cubic centimeter. For bulk and soil sample asbestos analysis EPA considers any material containing greater than 1% asbestos as asbestos containing material (ACM). Any material containing less than 1% asbestos is not considered ACM.

C. Quality Control

Overall quality control for the sampling phase of this project

shall be the joint responsibility of the TAT field team leader and the OSC. The laboratory quality control shall follow the quality control procedures specified by the respective methods.

VI. REFERENCES

Ecology & Environment, Inc., Technical Assistance Team, May 8, 1992. Certain-Teed Transite Pipe Site Assessment, TDD T07-9203-012, submitted to U.S. EPA Region VII Emergency Planning and Response Branch, Kansas City, Kansas.

Ecology & Environment, Inc., Technical Assistance Team, September 21, 1992. Certain-Teed-Maline Creek Site Assessment, TDD T07-9209-003, submitted to U.S. EPA Region VII Emergency Planning and Response Branch, Kansas City, Kansas.

Ecology & Environment, Inc., Technical Assistance Team, March 14, 1994. Maline Creek Site Assessment, TDD T07-9402-015, submitted to U.S. EPA Region VII Emergency Planning and Response Branch, Kansas City, Kansas.

Missouri Department of Natural Resources, Waste Management Unit, August 28, 1984, Preliminary Assessment-Branch Metal Processing Company, 3012 Summary, Case 534.918.

U.S. Environmental Protection Agency, 1988, Environmental Monitoring and Compliance Branch, Inspection Report on the Certain-Teed Transite Pipe Plant, St. Louis, Missouri.

U.S. Environmental Protection Agency, 1988, Environmental Monitoring and Compliance Branch, Inspection Report on the GAF Transite Plant, St. Louis, Missouri.

ATTACHMENTS

Attachment A - Site Location Map

Attachment B - Site Sketch

Attachment C - Areas Affected by Flooding/Proposed Sampling Locations

Attachment D - 29 CFR 1910.1001-Appendix A;B Sampling Procedures

ATTACHMENT A

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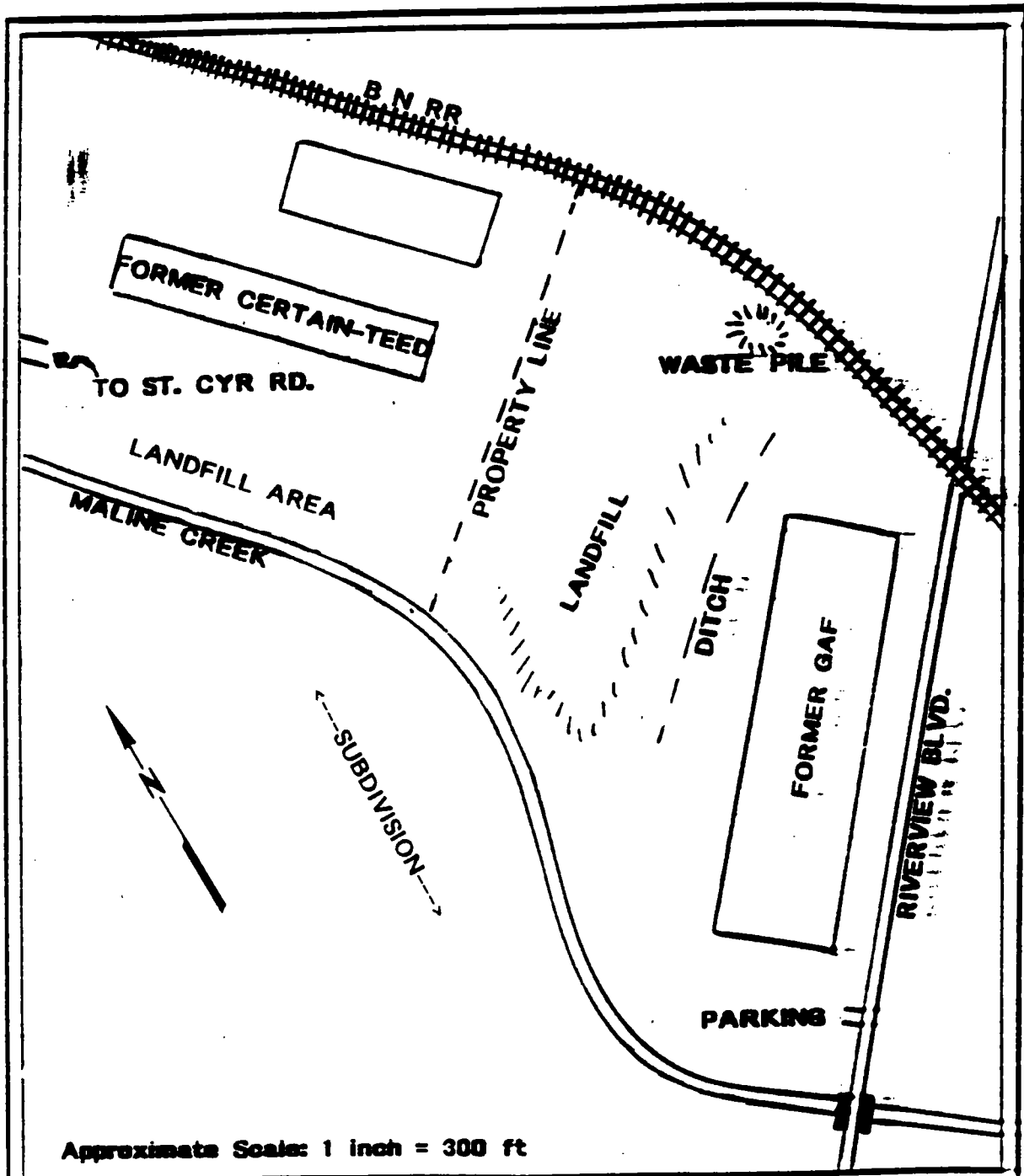
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ATTACHMENT B

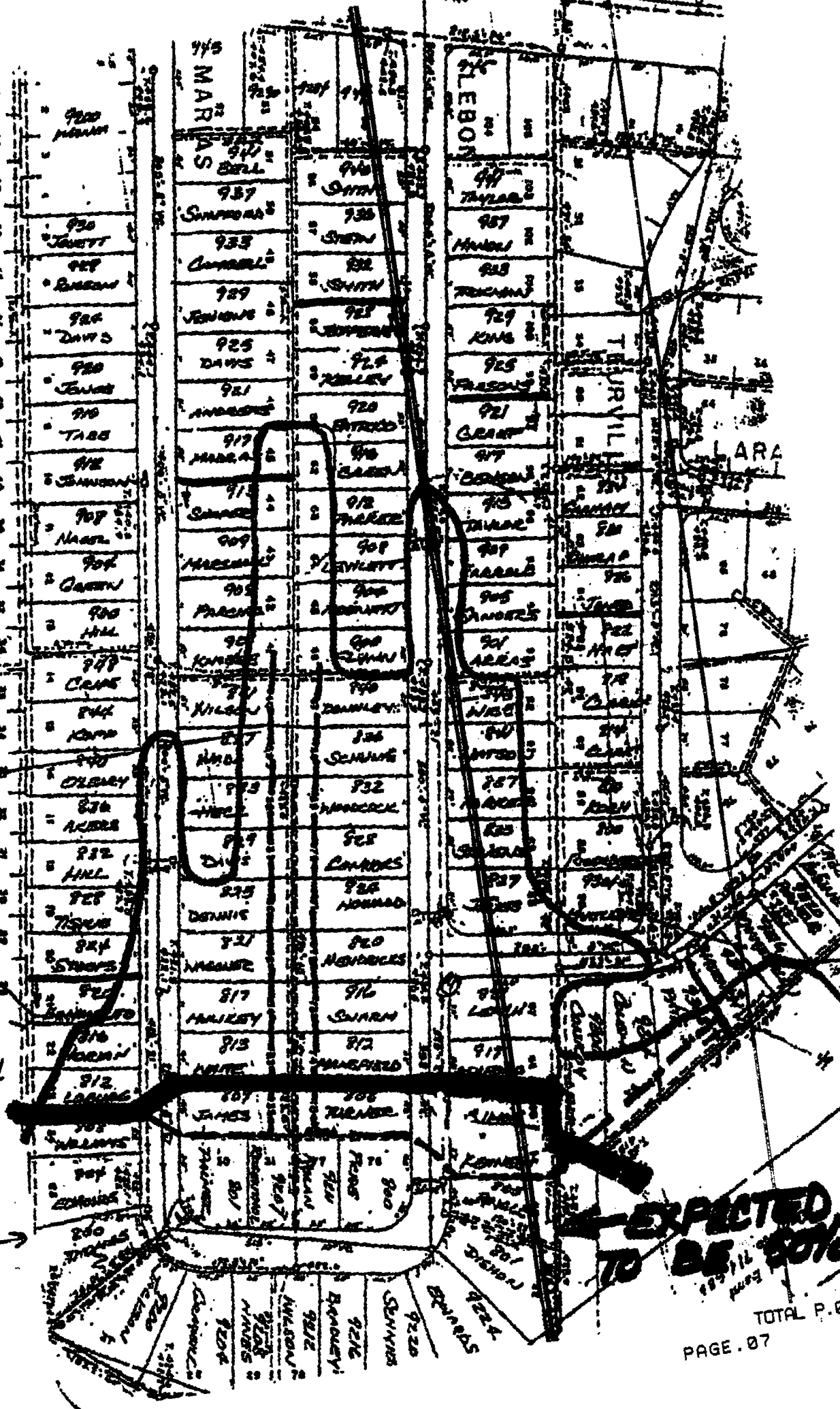


CERTAIN-TEED TRANSITE PIPE SITE (MALINE CREEK)

SITE MAP

ATTACHMENT C

TAINE
S.W.
PL.
BELLE CREST



area affected
by water

demolition
area

EXPECTED
TO BE 50%



ATTACHMENT D

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(iii) Paragraph (i) of this section, shall be complied with by September 14, 1989.

(4) *Compliance data.* The requirements of paragraphs (IX4), (JXIXIV), (JX5XIII1), (JX5XIIIJ), (JX5XIVXC), and (IX7XIXD) shall be complied with by May 7, 1990.

(p) *Appendices.* (1) Appendices A, C, D, and E to this section are incorporated as part of this section and the contents of these Appendices are mandatory.

(2) Appendices B, F, G, H, and I to this section are informational and are not intended to create any additional obligation not otherwise imposed or to detract from any existing obligation.

APPENDIX A to § 1910.1001—OSHA
Reference Method—Mandatory

This mandatory appendix specifies the procedure for analyzing air samples for asbestos and specifies quality control procedures that must be implemented by laboratories performing the analysis. The sampling and analytical methods described below represent the elements of the available monitoring methods (such as the NIOSH 7400 method) which OSHA considers to be essential to achieve adequate employee exposure monitoring while allowing employers to use methods that are already established within their organizations. All employers who are required to conduct air monitoring under paragraph (d) of the standard are required to utilize analytical laboratories that use this procedure, or an equivalent method, for collecting and analyzing samples.

Sampling and Analytical Procedure

1. The sampling medium for air samples shall be mixed cellulose ester filter membranes. These shall be designated by the manufacturer as suitable for asbestos counting. See below for rejection of blanks.

2. The preferred collection device shall be the 25-mm diameter cassette with an open-faced 30-mm electrically conductive exten-

sion bowl. The 37-mm cassette may be used if necessary but only if written justification for the need to use the 37-mm filter cassette accompanies the sample results in the employee's exposure monitoring record.

3. An air flow rate between 0.5 liter/min and 2.5 liters/min shall be selected for the 25-mm cassette. If the 37-mm cassette is used, an air flow rate between 1 liter/min and 2.5 liters/min shall be selected.

4. Where possible, a sufficient air volume for each air sample shall be collected to yield between 100 and 1,300 fibers per square millimeter on the membrane filter. If a filter darkens in appearance or if loose dust is seen on the filter, a second sample shall be started.

5. Ship the samples in a rigid container with sufficient packing material to prevent dislodging the collected fibers. Packing material that has a high electrostatic charge on its surface (e.g., expanded polystyrene) cannot be used because such material can cause loss of fibers to the sides of the cassette.

6. Calibrate each personal sampling pump before and after use with a representative filter cassette installed between the pump and the calibration device.

7. Personal samples shall be taken in the "breathing zone" of the employee (i.e., attached to or near the collar or lapel near the worker's face).

8. Fiber counts shall be made by positive phase contrast using a microscope with an 8 to 10 X eyepiece and a 40 to 45 X objective for a total magnification of approximately 400 X and a numerical aperture of 0.65 to 0.75. The microscope shall also be fitted with a green or blue filter.

9. The microscope shall be fitted with a Walton-Beckett eyepiece graticule calibrated for a field diameter of 100 micrometers (+/- 2 micrometers).

10. The phase-shift detection limit of the microscope shall be about 3 degrees measured using the HSE phase shift test slide as outlined below.

a. Place the test slide on the microscope stage and center it under the phase objective.

b. Bring the blocks of grooved lines into focus.

NOTE: The slide consists of seven sets of grooved lines (ca. 30 grooves to each block) in descending order of visibility from sets 1 to 7, seven being the least visible. The requirements for asbestos counting are that the microscope optics must resolve the grooved lines in set 3 completely, although they may appear somewhat faint, and that the grooved lines in sets 6 and 7 must be invisible. Sets 4 and 5 must be at least partially visible but may vary slightly in visibility between microscopes. A microscope that fails to meet these requirements has either

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too low or too high a resolution to be used for asbestos counting.

c. If the image deteriorates, clean and adjust the microscope optics. If the problem persists, consult the microscope manufacturer.

11. Each set of samples taken will include 10 percent blanks or a minimum of 3 blanks. The blank results shall be averaged and subtracted from the analytical results before reporting. Any samples represented by a blank having a fiber count in excess of 7 fibers/100 fields shall be rejected.

12. The samples shall be mounted by the acetone/triacetin method or a method with an equivalent index of refraction and similar clarity.

13. Observe the following counting rules.

a. Count only fibers equal to or longer than 5 micrometers. Measure the length of curved fibers along the curve.

b. In the absence of other information, count all particles as asbestos that have a length-to-width ratio (aspect ratio) of 3:1 or greater.

c. Fibers lying entirely within the boundary of the Walton-Beckett graticule field shall receive a count of 1. Fibers crossing the boundary once, having one end within the circle, shall receive the count of one half (1/2). Do not count any fiber that crosses the graticule boundary more than once. Reject and do not count any other fibers even though they may be visible outside the graticule area.

d. Count bundles of fibers as one fiber unless individual fibers can be identified by observing both ends of an individual fiber.

e. Count enough graticule fields to yield 100 fibers. Count a minimum of 20 fields; stop counting at 100 fields regardless of fiber count.

14. Blind recounts shall be conducted at the rate of 10 percent.

Quality Control Procedures

1. Intralaboratory program. Each laboratory and/or each company with more than one microscopist counting slides shall establish a statistically designed quality assurance program involving blind recounts and comparisons between microscopists to monitor the variability of counting by each microscopist and between microscopists. In a company with more than one laboratory, the program shall include all laboratories and shall also evaluate the laboratory-to-laboratory variability.

2. Interlaboratory program. Each laboratory analyzing asbestos samples for compliance determination shall implement an interlaboratory quality assurance program that as a minimum includes participation of at least two other independent laboratories. Each laboratory shall participate in round robin testing at least once every 6 months with at least all the other laboratories in its

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interlaboratory quality assurance group. Each laboratory shall submit slides typical of its own work load for use in this program. The round robin shall be designed and results analyzed using appropriate statistical methodology.

3. All individuals performing asbestos analysis must have taken the NIOSH course for sampling and evaluating airborne asbestos dust or an equivalent course.

4. When the use of different microscopes contributes to differences between counters and laboratories, the effect of the different microscope shall be evaluated and the microscope shall be replaced, as necessary.

5. Current results of these quality assurance programs shall be posted in each laboratory to keep the microscopists informed.

APPENDIX B TO § 1910.1001—DETAILED PROCEDURE FOR ASBESTOS SAMPLING AND ANALYSIS—NON-MANDATORY

This appendix contains a detailed procedure for sampling and analysis and includes those critical elements specified in Appendix A. Employers are not required to use this procedure, but they are required to use Appendix A. The purpose of Appendix B is to provide a detailed step-by-step sampling and analysis procedure that conforms to the elements specified in Appendix A. Since this procedure may also standardize the analysis and reduce variability, OSHA encourages employers to use this appendix.

Asbestos Sampling and Analysis Method

Technique: Microscopy, Phase Contrast

Analyte: Fibers (manual count)

Sample Preparation: Acetone/triacetin method

Calibration: Phase-shift detection limit about 3 degrees

Range: 100 to 1300 fibers/mm² filter area

Estimated limit of detection: 7 fibers/mm² filter area

Sampler: Filter (0.8-1.2 um mixed cellulose ester membrane, 25-mm diameter)

Flow rate: 0.5 L/min to 2.5 L/min (25-mm cassette) 1.0 L/min to 2.5 L/min (37-mm cassette)

Sample volume: Adjust to obtain 100 to 1300 fibers/mm²

Shipment: Routine

Sample stability: Indefinite

Blank: 10% of samples (minimum 2)

Standard analytical error: 0.25.

Applicability: The working range is 0.02 f/cc (1920-L air sample) to 1.25 f/cc (480-L air sample). The method gives an index of air-

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boring asbestos fibers but may be used for other materials such as fibrous glass by inserting suitable parameters into the counting rules. This method does not differentiate between asbestos and other fibers. Asbestos fibers less than ca. 0.25 um diameter will not be detected by this method.

Interference: Any other airborne fiber may interfere since all particles meeting the counting criteria are counted. Chalklike particles may appear fibrous. High levels of nonfibrous dust particles may obscure fibers in the field of view and raise the detection limit.

Reagents: 1. Acetone. 2. Tritonin (glycerol triacetate), reagent grade.

Special precautions: Acetone is an extremely flammable liquid and precautions must be taken not to ignite it. Heating of acetone must be done in a ventilated laboratory fume hood using a flameless, spark-free heat source.

Equipment: 1. Collection device: 25-mm cassette with 50-mm electrically conductive extension (owl) with cellulose ester filter, 0.8 to 1.3 um pore size and backup pad.

Notes: Analyze representative filters for fiber background before use and discard the filter lot if more than 5 fibers/100 fields are found.

2. Personal sampling pump, greater than or equal to 0.5 L/min, with flexible connecting tubing.

3. Microscope, phase contrast, with green or blue filter, 8 to 10X eyepiece, and 40 to 45X phase objective (total magnification ca 400X; numerical aperture = 0.68 to 0.78).

4. Slide, glass, single-throw, pre-cleaned, 25 x 75 mm.

5. Cover slip, 25 x 25 mm, no. 1 1/4 unless otherwise specified by microscope manufacturer.

6. Knife, No. 1 surgical steel, curved blade.

7. Tweezers.

8. Flask, Guth-type, insulated neck, 250 to 500 mL (with single-holed rubber stopper and elbow-jointed glass tubing, 18 to 22 cm long).

9. Hotplate, spark-free, stirring type; heating mantle, or infrared lamp and magnetic stirrer.

10. Syringe, hypodermic, with 32-gauge needle.

11. Graticule, Walton-Beckett type with 100 um diameter circular field at the specimen plane (area = 0.00785 mm², (Type G-23).

NOTE: the graticule is custom-made for each microscope.

12. HSE/NPL phase contrast test slide, Mark II.

13. Telescope, ocular phase-ring centering.

14. Stage micrometer (0.01 mm divisions).

Sampling

1. Calibrate each personal sampling pump with a representative sampler in lab.

2. Fasten the sampler to the worker's lapel as close as possible to the worker's mouth. Remove the top cover from the end of the owl extension (open face) and orient face down. Wrap the joint between the extender and the monitor's body with shrink tape to prevent air leaks.

3. Submit at least two blanks (or 10% of the total samples, whichever is greater) for each set of samples. Remove the caps from the field blank cassettes and store the caps and cassettes in a clean area (bag or box) during the sampling period. Replace the caps in the cassettes when sampling is completed.

4. Sample at 0.5 L/min or greater. Do not exceed 1 mg total dust loading on the filter. Adjust sampling flow rate, Q (L/min), and time to produce a fiber density, E (fibers/mm²), of 100 to 1500 fibers/mm² (3.65 x 10⁵ to 5 x 10⁶ fibers per 25-mm filter with effective collection area (A_e = 348 mm²) for optimum counting precision (see step 21 below). Calculate the minimum sampling time, t_{min} (min) at the action level (one-half of the current standard), L (L/cc) of the fibers being sampled:

$$t_{min} = \frac{(AC)(E)}{(Q)(L)10^6}$$

5. Remove the field monitor at the end of sampling, replace the plastic top cover and small end caps, and store the monitor.

6. Ship the samples in a rigid container with sufficient packing material to prevent jostling or damage.

NOTE: Do not use polycarbonate foam in the shipping container because of electrostatic forces which may cause fiber loss from the sample filter.

Sample Preparation

NOTE: The object is to produce samples with a smooth (non-grainy) background in a medium with a refractive index equal to or less than 1.46. The method below collapses the filter for easier focusing and produces permanent mounts which are useful for quality control and interlaboratory comparison. Other mounting techniques meeting the above criteria may also be used, e.g., the nonpermanent field mounting technique used in P & CAM 230.

7. Insure that the glass slides and cover slips are free of dust and fibers.

8. Place 40 to 60 mL of acetone into a Guth-type flask. Stopper the flask with a single-hole rubber stopper through which a glass tube extends 5 to 8 cm into the flask.

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The portion of the glass tube that exits the top of the stopper (8 to 10 cm) is bent downward in an elbow that makes an angle of 20 to 30 degrees with the horizontal.

9. Place the flask in a stirring hotplate or wrap in a heating mantle. Heat the acetone gradually to its boiling temperature (ca. 58 °C).

CAUTION.—The acetone vapor must be generated in a ventilated fume hood away from all open flames and spark sources. Alternate heating methods can be used, providing no open flame or sparks are present.

10. Mount either the whole sample filter or a wedge cut from the sample filter on a clean glass slide.

a. Cut wedges of ca. 25 percent of the filter area with a curved-blade steel surgical knife using a rocking motion to prevent tearing.

b. Place the filter or wedge, dust side up, on the slide. Static electricity will usually keep the filter on the slide until it is cleared.

c. Hold the glass slide supporting the filter approximately 1 to 2 cm from the glass tube port where the acetone vapor is escaping from the heated flask. The acetone vapor stream should cause a condensation spot on the glass slide ca. 2 to 3 cm in diameter. Move the glass slide gently in the vapor stream. The filter should clear in 2 to 5 min. If the filter curls, distorts, or is otherwise rendered unusable, the vapor stream is probably not strong enough. Periodically wipe the outlet port with tissue to prevent liquid acetone dripping onto the filter.

d. Using the hypodermic syringe with a 22-gauge needle, place 1 to 2 drops of triacetin on the filter. Gently lower a clean 28-mm square cover slip down onto the filter at a slight angle to reduce the possibility of forming bubbles. If too many bubbles form or the amount of triacetin is insufficient, the cover slip may become detached within a few hours.

e. Glue the edges of the cover slip to the glass slide using a lacquer or nail polish.

NOTE: If clearing is slow, the slide preparation may be heated on a hotplate (surface temperature 50 °C) for 15 min to hasten clearing. Counting may proceed immediately after clearing and mounting are completed.

Calibration and Quality Control

11. Calibration of the Walton-Beckett graticule. The diameter, d (mm), of the circular counting area and the disc diameter must be specified when ordering the graticule.

a. Insert any available graticule into the eyepiece and focus so that the graticule lines are sharp and clear.

b. Set the appropriate interpupillary distance and, if applicable, reset the binocular

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head adjustment so that the magnification remains constant.

c. Install the 40 to 45X phase objective.

d. Place a stage micrometer on the microscope object stage and focus the microscope on the graduated lines.

e. Measure the magnified grid length, L_s (mm), using the stage micrometer.

f. Remove the graticule from the microscope and measure its actual grid length, L_a (mm). This can best be accomplished by using a stage fitted with verniers.

g. Calculate the circle diameter, d (mm), for the Walton-Beckett graticule:

$$d_s = \frac{L_s \times D}{L_a}$$

EXAMPLE.—If $L_s = 108$ um, $L_a = 2.93$ mm and $D = 100$ um, then $d_s = 2.71$ mm.

h. Check the field diameter, D (acceptable range 100 mm \pm 2 mm) with a stage micrometer upon receipt of the graticule from the manufacturer. Determine field area (mm²).

12. Microscope adjustments. Follow the manufacturer's instructions and also the following:

a. Adjust the light source for even illumination across the field of view at the condenser iris.

NOTE: Kohler illumination is preferred, where available.

b. Focus on the particulate material to be examined.

c. Make sure that the field iris is in focus, centered on the sample, and open only enough to fully illuminate the field of view.

d. Use the telescope ocular supplied by the manufacturer to ensure that the phase rings (annular diaphragm and phase-shifting elements) are concentric.

13. Check the phase-shift detection limit of the microscope periodically.

a. Remove the HSE/NPL phase-contrast test slide from its shipping container and center it under the phase objective.

b. Bring the blocks of grooved lines into focus.

NOTE: The slide consists of seven sets of grooves (ca. 20 grooves to each block) in descending order of visibility from sets 1 to 7. The requirements for counting are that the microscope optics must resolve the grooved lines in set 3 completely, although they may appear somewhat faint, and that the grooved lines in sets 6 to 7 must be invisible. Sets 4 and 5 must be at least partially visible but may vary slightly in visibility between microscopes. A microscope which fails to meet these requirements has either too low

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or too high a resolution to be used for asbestos counting.

c. If the image quality deteriorates, clean the microscope optics and, if the problem persists, consult the microscope manufacturer.

14. Quality control of fiber counts.

a. Prepare and count field blanks along with the field samples. Report the counts on each blank. Calculate the mean of the field blank counts and subtract this value from each sample count before reporting the results.

NOTE 1: The identity of the blank filters should be unknown to the counter until all counts have been completed.

NOTE 2: If a field blank yields fiber counts greater than 7 fibers/100 fields, report possible contamination of the samples.

b. Perform blind recounts by the same counter on 10 percent of filters counted (slides relabeled by a person other than the counter).

15. Use the following test to determine whether a pair of counts on the same filter should be rejected because of possible bias. This statistic estimates the counting repeatability at the 95% confidence level. Discard the sample if the difference between the two counts exceeds $2.77(F/\bar{P})$, where \bar{P} = average of the two fiber counts and s = relative standard deviation, which should be derived by each laboratory based on historical in-house data.

NOTE: If a pair of counts is rejected as a result of this test, recount the remaining samples in the set and test the new counts against the first counts. Discard all rejected paired counts.

16. Enroll each new counter in a training course that compares performance of counters on a variety of samples using this procedure.

NOTE: To ensure good reproducibility, all laboratories engaged in asbestos counting are required to participate in the Proficiency Analytical Testing (PAT) Program and should routinely participate with other asbestos fiber counting laboratories in the exchange of field samples to compare performance of counters.

Measurement

17. Place the slide on the mechanical stage of the calibrated microscope with the

center of the filter under the objective lens. Focus the microscope on the plane of the filter.

18. Regularly check phase-ring alignment and Kohler illumination.

19. The following are the counting rules:

a. Count only fibers longer than 5 μ m. Measure the length of curved fibers along the curve.

b. Count only fibers with a length-to-width ratio equal to or greater than 3:1.

c. For fibers that cross the boundary of the graticule field, do the following:

1. Count any fiber longer than 5 μ m that lies entirely within the graticule area.

2. Count as $\frac{1}{2}$ fiber any fiber with only one end lying within the graticule area.

3. Do not count any fiber that crosses the graticule boundary more than once.

4. Reject and do not count all other fibers.

d. Count bundles of fibers as one fiber unless individual fibers can be identified by observing both ends of a fiber.

e. Count enough graticule fields to yield 100 fibers. Count a minimum of 20 fields. Stop at 100 fields regardless of fiber count.

20. Start counting from one end of the filter and progress along a radial line to the other end, shift either up or down on the filter, and continue in the reverse direction. Select fields randomly by looking away from the eyepiece briefly while advancing the mechanical stage. When an agglomerate covers ca. $\frac{1}{4}$ or more of the field of view, reject the field and select another. Do not report rejected fields in the number of total fields counted.

NOTE: When counting a field, continuously scan a range of focal planes by moving the fine focus knob to detect very fine fibers which have become embedded in the filter. The small-diameter fibers will be very faint but are an important contribution to the total count.

Calculations

21. Calculate and report fiber density on the filter, E (fibers/mm²), by dividing the total fiber count, F , minus the mean field blank count, B , by the number of fields, n ; and the field area, A , (0.00785 mm² for a properly calibrated Walton-Beckett graticule):

$$E = \frac{(F/n) - (B/n_b)}{A_f} \text{ fibers/mm}^2$$

where:

n = number of fields in submission sample

n_b = number of fields in blank sample

SKC Guide to NIOSH/OSHA Air Sampling Standards

			S A M P L I N G												
Chemical Hazard	Agency	Ref	Agency Standard		Vol. (liter)		Rate (ml/min)		Time		Analytical Method	SKC Collecting Equipment and Page Number			
			TWA (ppm)	Ceiling (ppm)	TWA	Ceiling	TWA	Ceiling	TWA (hrs)	Ceiling (min)					
Aluminum, Alkyls	OSHA	IMISA100	2 mg/m ³		960		2000		8		AAS	FLT 225-8-01	31	HLD 225-3	31
Aluminum & Compounds (as Al)	NIOSH	7013			360		1000		6		AAS-F	FLT 225-5	31	HLD 225-3	31
Aluminum, Metal & Oxide	OSHA	IMISA100	10 mg/m ³		960		2000		8		AAS	FLT 225-8-01	31	HLD 225-3	31
Aluminum Oxide	OSHA	IMIS0160	10 mg/m ³		960		2000		8		AAS	FLT 225-8-01	31	HLD 225-3	31
Aluminum, Pyro Powders	OSHA	IMISA100	5 mg/m ³		960		2000		8		AAS	FLT 225-8-01	31	HLD 225-3	31
Aluminum, Soluble Salts	OSHA	IMISA100	2 mg/m ³		960		2000		8		AAS	FLT 225-8-01	31	HLD 225-3	31
Aluminum, Welding Fumes	OSHA	IMISA100	5 mg/m ³		960		2000		8		AAS	FLT 225-8-01	31	HLD 225-3	31
Amines, Aliphatic	NIOSH	2010	varies		20		40		8		GC-FID	ST 226-10	7		
Amines, Aromatic	NIOSH	2002	varies		20		40		8		GC-FID	ST 226-10	7		
p-Amino Acetanilide	OSHA	IMIS0161									NVM				
p-Amino Azobenzene	OSHA	IMISA508									NVM				
4-Amino Biphenyl	NIOSH	4(269)			48		200		4		GC	FLT 225-16 ST 226-47	31	HLD 225-32	31
4-Amino Diphenyl	OSHA	IMIS0182			50		1000		50min		HPLC-UV	IMP 225-36-1	29	IT 225-22	29
2-Amino Ethanol	NIOSH	2007			10		20		8		GC-FID	ST 226-10-04	7		
p-Amino Phenylarsonic Acid	NIOSH	5022			960		2000		8		IC-AAS	FLT 225-17-01	31	HLD 225-3	31
bis-2-Amino Propyl Ether	OSHA	IMIS0184									NVM				
2-Amino Pyridine	NIOSH	4(S158)	0.5		12		100		2		GC	ST 226-35-02	7		
2-Amino Pyridine	OSHA	IMIS0185	0.5		10		20(50)		8(3.3)		GC-FID-W	ST 226-36 (2)	7		
3-Amino-1-Propanol	OSHA	IMISA608									NVM				
1-Amino-2-Propanol	OSHA	IMISA608									NVM				
2-Aminoethanol	NIOSH	3509			240		1000		4		IC	IMP 225-36-1	29	IT 225-22	29
Aminoethanol Compounds II	NIOSH	3509			240		1000		4		IC	IMP 225-361	29	IT 225-22	29
Amitrole	OSHA	IMISA176	0.2 mg/m ³								NVM				
Ammonia		18			18	2.5	75	500	4	5	CLR	ST 226-61	7		
Ammonia	NIOSH	1(205)		50/5min	15		1000		15min		CLR	IMP 225-36-1	29	IT 225-22	29
Ammonia	NIOSH	5(S347)		50/5min	30		200		8		SPI ELEC	ST 226-10-08	7	FLT 225-5	31
												HLD 225-2	31	SCN 225-26	34
Ammonia	NIOSH	6701		50/5min							IC	BDGC 540-02	44		
Ammonia	OSHA	ID 188	35 (STEL)		1.5		100		15min		IC	ST PENDING			
Ammonium Chloride	OSHA	ID 188	10 mg/m ³ *		24		100		4		IC	ST PENDING			
Ammonium Hydroxide (see Ammonia)															
Ammonium Nitrate	OSHA	IMISA613									NVM				
Ammonium Sulfamate	NIOSH	5(S348)	15 mg/m ³		90		1500		1		IC	FLT 225-5	31	HLD 225-3	31
Ammonium Sulfamate (Respirable Dust)	OSHA	IMIS0185	5 mg/m ³		912		1900		8		GR	FLT 225-8-01 F/HLD 225-1	31	HLD 225-3 CYC 225-01-02	31 33
Ammonium Sulfamate (Total Dust)	OSHA	IMIS0185	10 mg/m ³		720		1500		8		GR	FLT 225-8-01	31	HLD 225-3	31
n- & secAmyl Acetate	NIOSH	1450			10		20(50)		8(3.3)		GC-FID	ST 226-01	7		
n-Amyl Acetate	OSHA	07-A	100		10		20(50)		8(3.3)		GC-FID	ST 226-01	7		
sec-Amyl Acetate	OSHA	07-B	125		10		20(50)		8(3.3)		GC-FID	ST 226-01	7		
Amyl Nitrite	OSHA	IMISA607			8		50		2.5		HPLC-UV	ST 226-01	7		
Aniline	NIOSH	2002			20		40		8		GC-FID	ST 226-10	7		
Aniline	NIOSH	3(S310)			20		40		8		GC	ST 226-10	7		
Aniline	OSHA	IMIS0220	2		20		40		8		GC-FID	ST 226-10	7		
Anisidine	NIOSH	2514			240		1000		4		HPLC-UV	ST 226-30-05	7		
Anisidine (o,p, isomers)	NIOSH	5(S163)			240		500		8		HPLC	ST 226-30-05	7		
Anisidine (o,p, isomers)	OSHA	IMIS0225	0.5 mg/m ³		240		1000		4		HPLC	ST 226-30	7		
Anthanthrene	NIOSH	1(183)			600		2000		5		GC-CLR	FLT 225-7 HLD 225-3	31	FLT 225-1801	31
Anthrophyllite Fibers	NIOSH	7400	0.1 f/ml		960		2000		8		PCM			FL/CL 225-3-18 or FL/CL 225-3-20	32 32
Anthracene	OSHA	58	0.2 mg/m ³		960		2000		8		GR HPLC	FLT 225-7	31	HLD 225-2	31
Antimony & Compounds (as Sb)	NIOSH	2(S2)			360		1500		4		AA	FLT 225-5	31	HLD 225-3	31
Antimony & Compounds (as Sb)	OSHA	ID 121	0.5 mg/m ³		960		2000		8		ICPAES	FLT 225-5	31	HLD 225-3	31
Antimony & Compounds (as Sb)	OSHA	ID 125	0.5 mg/m ³		960		2000		8		AAS-W	FLT 225-5	31	HLD 225-3	31
Antimony Particulates	NIOSH	4(261)			45		1500		30min		AA	FLT 225-5	31	HLD 225-3	31
Antineoplastic Drugs	OSHA	IMISA617									NVM				
Antu (Alphanaphthyl Thiourea)	NIOSH	5(S276)			480		2000		4		HPLC	FLT 225-17-01	31	HLD 225-2	31
Antu (Alphanaphthyl Thiourea)	OSHA	IMIS0235	0.3 mg/m ³		480		2000		8		HPLC-UV	FLT 225-17-01	31	HLD 225-2	31
Aqua Fortis	NIOSH	7903	2		48		200		4		IC	ST 226-10-03	7		
Argon	OSHA	IMIS0240									NVM				
Arsenic	NIOSH	7300		2 ug/m ³	960		2000		8		ICPAES	FLT 225-5	31	HLD 225-3	31
Arsenic	OSHA	ID 105	.01 mg/m ³		960		2000		8		AAS-GF	FLT 225-5	31	HLD 225-2	31
Arsenic & Compounds (as As)	NIOSH	7900		2 ug/m ³ /15	30		2000		15		AA, FLARGN	FLT 225-5	31	HLD 225-3	31
Arsenic & Compounds (as As)	OSHA	IMIS0260	.01 mg/m ³		480		1000		8		AAS-GF, W	FLT 225-5 SM TB 225-24	31	HLD 225-3	31
Arsenic Organo	NIOSH	5022			96		2000		8		IC-AAS	FLT 225-17-01	31	HLD 225-2	31
Arsenic Trioxide (as As)	NIOSH	7901		2 ug/m ³ /15	30		2000		15		AAS-GF	FLT 225-5	31	HLD 225-3	31
Arsenicate Particulates	NIOSH	6(320)			300		1500		3.3		IC-AAS	FLT 225-17-01	31	HLD 225-3	31
Arsine	NIOSH	6001			10		20		8		AA-GF	ST 226-01	7		
Arsine	OSHA	IMIS0270	0.05		30		200		2.5		AAS/GF	ST 226-01	7		
Arylam (see Carbaryl)															
Asbestos	OSHA	ID 160	0.2 fbr/cc		240		2000		2		PCM			FLT 225-3-12	31
Asbestos Fibers	NIOSH	7400	0.1 fbr/cc		960		2000		8		PCM			FLT 225-3-12 or FLT 225-3-20	31 31
Asbestos Fibers	NIOSH	7402	0.1 fbr/cc		960		2000		8		TEM	FLT 225-3-12	31		

VERS April 27, 1993

NM Asbestos (all forms)

DESC Fine, slender, flaxy fibers; resists fire and most solvents.

IMIS 9020

CAS 1332-21-4

NIOSH RTECS C16475000

DOT 2212 31; 2590 31

OSHA Cancer & Lung Disease Hazard 29 CFR 1910.1001

. TWA 0.2 F/cc

. STEL 1.0 F/cc

. ACTION LEVEL 0.1 F/cc

TLV See Dusts and Appendix A1a (Human carcinogens)

REL 0.1 fiber/cc per 400 L air sample; Carcinogen

IARC Group 1, carcinogenic to humans

NTP Human Carcinogen

HLTH Cancer (HE1); Asbestosis (HE10)

SYMPT Dyspnea; interstitial fibrosis; restricted pulmonary functioning;
finger clubbing; (carcinogenic)

ORG Lungs

SLC1 MEDIA: Mixed Cellulose Ester Filter (MCEF) 0.8 microns (open face)

. 25 mm cassette with 50 mm conductive cowl

MAX V: 1200 Liters MAX F: 2.5 L/min MIN F: 0.5 L/min (TWA)

MIN V: 48 Liters MAX F: 2.5 L/min MIN F: 1.6 L/min (STEL)

ANL 1: Phase Contrast Microscopy; PCM

. REF: 2 (OSHA ID-160) SAE: 0.25 CLASS: Fully Validated

NOTE: Do not request multiple analytes. Do not overload. If dust
is high, reduce air volume to avoid overloading. A minimum of 2
blanks or 10% are required for every set.

WIPE Do not use Whatman or other paper filters. Bulk preferred.

DIV J

BRANCH MIC